organic compounds

4-(4-Methoxyphenethyl)-3-methyl-1H-1,2,4-triazol-5(4H)-one

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The dihedral angle between the two rings in the title compound, $C_{12}H_{15}N_3O_2$, is 49.03 (1)°. The crystal structure is stabilized by intermolecular N—H···O and C—H···O hydrogen bonds and π–π stacking interactions between the triazole rings with a centroid–centroid distance of 3.394 Å.

Related literature

For related literature on triazole compounds, see: Tanak *et al.* (2010); Ünver *et al.* (2008); Ünver, Düğdü *et al.* (2009); Ünver, Sancak *et al.* (2009). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).
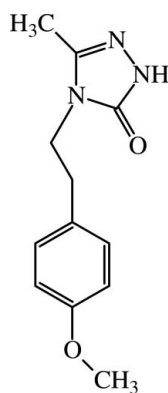
Experimental

Crystal data

C_{12}H_{15}N_3O_2 ... M_r = 233.27

Monoclinic, $P2_1/c$
a = 14.7736 (9) Å
b = 5.6986 (2) Å
c = 15.2478 (9) Å
β = 110.726 (5)°
V = 1200.62 (11) Å^3

Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
$0.80 \times 0.40 \times 0.13$ mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.955, T_{max} = 0.987

8004 measured reflections
3000 independent reflections
1699 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.107
S = 0.90
3000 reflections
160 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.10 e Å^{-3}
$\Delta\rho_{min}$ = −0.10 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O2^i	0.969 (17)	1.847 (18)	2.8068 (18)	170.2 (14)
C8—H8B···O2^{ii}	0.97	2.57	3.3260 (18)	135

Symmetry codes: (i) $-x, -y+3, -z$; (ii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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supporting information

4-(4-Methoxyphenethyl)-3-methyl-1*H*-1,2,4-triazol-5(4*H*)-one

Yavuz Köysal, Hasan Tanak, Dilek Ünlüer and Şamil Işık

S1. Comment

1,2,4-Triazoles are an important class of heterocycles, and have been the subject of great interest due to their pharmacological properties (Ünver *et al.*, 2008; Ünver, Düğdü *et al.*, 2009; Ünver, Sancak *et al.*, 2009). 1,2,4-Triazole and 1,2,4- triazol-3-one are reported to exhibit a broad spectrum of biological activities such as antifungal, antimicrobial, hypoglycemic, antihypertensive, antidepressant, plant growth regulator anticoagulant, analgesic, antiparasitic, antiviral, anti-inflammatory, antitumor and anti-HIV properties (Tanak *et al.*, 2010).

In the title compound, triazol ring is oriented with respect to the methoxyphenethyl ring at dihedral angles of 49.03 (1)°, that shows, whole molecule is not planar. Triazol ring system is almost planar with the maximum deviation of -0.005 (1)Å for atom C11. The double bond distance in the triazol group is good agreement with our previous report,5-benzyl-4-(3,4-dimethoxyphenethyl)-2*H*-1,2,4-triazol-3(4*H*)-one (Tanak *et al.*, 2010).

Structurally, the title compound contains intermolecular N—H···O and C—H···O type hydrogen bonds, namely N3—H3···O2 (symmetry code:-*x*,3 - *y*,-*z*) which generates eight-membered ring, producing a $R_2^2(8)$ motif (Bernstein *et al.*, 1995) and C8—H8B···O2 (symmetry code:-*x*,*y* - 1/2,-*z* + 1/2) which generates twelve-membered ring, producing a $R_2^2(12)$ motif (Bernstein *et al.*, 1995) where atom O2 accepts hyrogen bonds from different donors. There is also π-π stacking interaction between the parallel triazol systems. The closest perpendicular distance is 3.394Å between the ring centroids at (*x*,*y*,*z*) and that at (-*x*,2 - *y*,-*z*). The details of the hydrogen bond is shown in Table 1.

S2. Experimental

Ethyl 2-[1-ethoxy-2-(phenyl)ethylidene]hydrazine carboxylate (10 mmol) together with 2-(4 -methoxyphenyl)ethylamin (10 mmol) were heated without solvent in a sealed tube for 2 h at 423- 433°K. Then, the mixture was cooled to r.t. and a solid cure formed. The crude product was recrystallized using ethyl acetate/petroleum ether (1:1) to afford the desired compound. Yield: 185 mg (82%). Mp: 416°K

S3. Refinement

The H atom bonded to N was refined isotropically. Other H atoms were refined using a riding model, with C—H distances ranging from 0.93–0.97 Å and U(H) set to $1.2U_{eq}(C)$ or $1.5U_{eq}(C_{methyl})$.
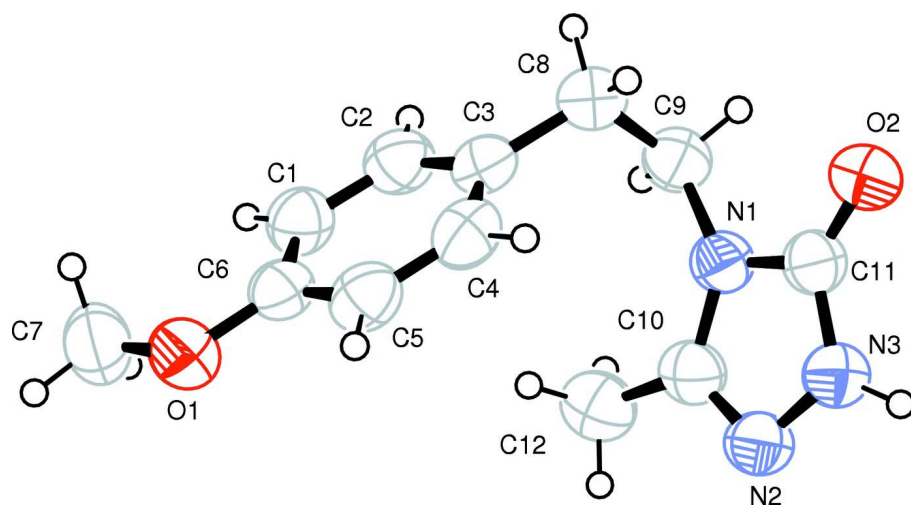
Figure 1

A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids.
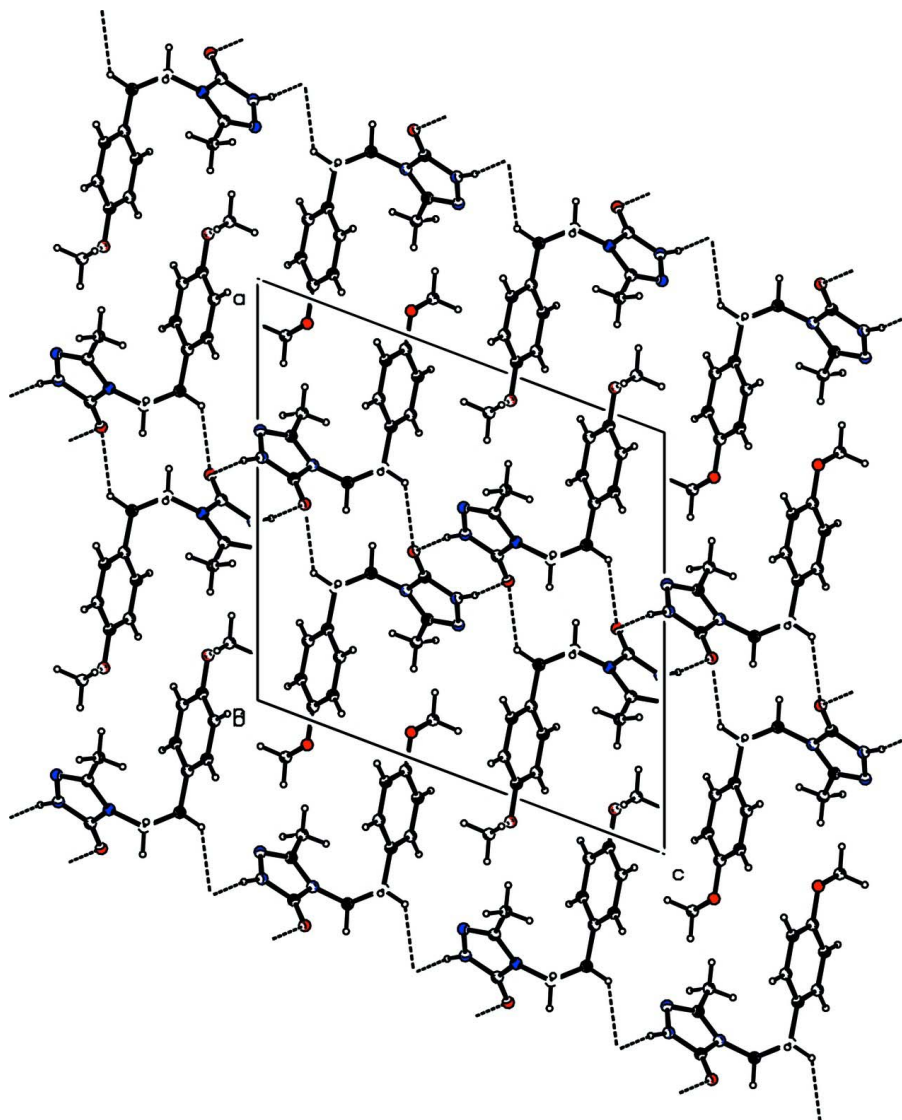
Figure 2

A partial packing view of (I).

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Crystal data

$C_{12}H_{15}N_3O_2$	$F(000) = 496$
$M_r = 233.27$	$D_x = 1.291$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 5937 reflections
$a = 14.7736$ (9) Å	$\theta = 1.5–28.7°$
$b = 5.6986$ (2) Å	$\mu = 0.09$ mm^{-1}
$c = 15.2478$ (9) Å	$T = 293$ K
$\beta = 110.726$ (5)°	PRISM., colourless
$V = 1200.62$ (11) Å^3	$0.80 \times 0.40 \times 0.13$ mm
$Z = 4$	

Data collection

Stoe IPDS 2 diffractometer	8004 measured reflections
Radiation source: fine-focus sealed tube	3000 independent reflections
Graphite monochromator	1699 reflections with $I > 2\sigma(I)$
Detector resolution: 6.67 pixels mm^{-1}	$R_{int} = 0.038$
rotation method scans	$\theta_{max} = 28.4°$, $\theta_{min} = 1.5°$
Absorption correction: integration	$h = -19 \rightarrow 19$
(*X-RED32*; Stoe & Cie, 2002)	$k = -7 \rightarrow 6$
$T_{min} = 0.955$, $T_{max} = 0.987$	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.039$	
$wR(F^2) = 0.107$	$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2]$
$S = 0.90$	where $P = (F_o^2 + 2F_c^2)/3$
3000 reflections	$(\Delta/\sigma)_{max} < 0.001$
160 parameters	$\Delta\rho_{max} = 0.10$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.10$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.017 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O2	0.00721 (8)	1.3809 (2)	0.11587 (7)	0.0781 (3)
C12	0.22054 (12)	0.7523 (3)	0.10928 (12)	0.0800 (5)
H12A	0.2410	0.7090	0.0583	0.120*
H12B	0.1862	0.6238	0.1237	0.120*
H12C	0.2762	0.7895	0.1634	0.120*
H3	0.0546 (12)	1.377 (3)	−0.0366 (12)	0.083 (5)*
O1	0.56139 (8)	0.9311 (2)	0.37786 (7)	0.0809 (3)
N1	0.10666 (8)	1.0519 (2)	0.13533 (7)	0.0607 (3)
N2	0.14153 (9)	1.0787 (2)	0.00638 (8)	0.0685 (3)
N3	0.07981 (9)	1.2572 (3)	0.01078 (8)	0.0676 (3)
C10	0.15659 (10)	0.9585 (3)	0.08233 (10)	0.0631 (3)
C3	0.26905 (10)	1.0401 (3)	0.32804 (8)	0.0626 (4)
C6	0.46536 (10)	0.9552 (3)	0.36347 (9)	0.0614 (4)
C4	0.32447 (11)	1.1936 (3)	0.29816 (9)	0.0680 (4)

H4	0.2958	1.3284	0.2658	0.082*
C5	0.42110 (12)	1.1528 (3)	0.31485 (9)	0.0681 (4)
H5	0.4565	1.2586	0.2933	0.082*
C2	0.31463 (11)	0.8424 (3)	0.37550 (10)	0.0689 (4)
H2	0.2789	0.7351	0.3959	0.083*
C11	0.05789 (9)	1.2457 (3)	0.08940 (9)	0.0626 (4)
C1	0.41146 (11)	0.7983 (3)	0.39378 (10)	0.0669 (4)
H1	0.4402	0.6637	0.4263	0.080*
C9	0.09688 (11)	0.9628 (3)	0.22112 (10)	0.0720 (4)
H9A	0.1107	0.7959	0.2259	0.086*
H9B	0.0305	0.9835	0.2174	0.086*
C8	0.16332 (12)	1.0831 (3)	0.30847 (10)	0.0790 (4)
H8A	0.1513	1.2507	0.3023	0.095*
H8B	0.1476	1.0292	0.3617	0.095*
C7	0.61047 (13)	0.7352 (4)	0.43098 (13)	0.0942 (5)
H7A	0.6771	0.7382	0.4359	0.141*
H7B	0.5807	0.5930	0.4004	0.141*
H7C	0.6068	0.7417	0.4925	0.141*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0736 (7)	0.0959 (8)	0.0722 (6)	0.0141 (6)	0.0348 (5)	0.0035 (5)
C12	0.0804 (11)	0.0778 (10)	0.0872 (10)	−0.0004 (9)	0.0364 (9)	−0.0069 (9)
O1	0.0652 (7)	0.1013 (8)	0.0781 (6)	0.0007 (6)	0.0277 (5)	−0.0057 (6)
N1	0.0515 (6)	0.0738 (7)	0.0575 (6)	−0.0071 (6)	0.0203 (5)	−0.0038 (6)
N2	0.0627 (7)	0.0826 (9)	0.0634 (7)	−0.0042 (6)	0.0262 (6)	−0.0073 (6)
N3	0.0612 (7)	0.0832 (9)	0.0598 (6)	0.0005 (6)	0.0234 (5)	0.0000 (6)
C10	0.0552 (7)	0.0703 (9)	0.0651 (8)	−0.0129 (7)	0.0229 (6)	−0.0105 (7)
C3	0.0690 (9)	0.0723 (9)	0.0469 (6)	0.0027 (7)	0.0210 (6)	−0.0041 (6)
C6	0.0626 (8)	0.0706 (9)	0.0509 (6)	−0.0029 (7)	0.0202 (6)	−0.0092 (7)
C4	0.0789 (10)	0.0640 (9)	0.0562 (7)	0.0023 (8)	0.0179 (7)	0.0030 (6)
C5	0.0757 (10)	0.0693 (9)	0.0589 (7)	−0.0114 (8)	0.0232 (7)	0.0026 (7)
C2	0.0744 (10)	0.0740 (9)	0.0622 (8)	−0.0060 (8)	0.0290 (7)	0.0044 (7)
C11	0.0503 (7)	0.0789 (10)	0.0575 (7)	−0.0076 (7)	0.0177 (6)	−0.0055 (7)
C1	0.0740 (9)	0.0626 (9)	0.0637 (8)	0.0061 (7)	0.0241 (7)	0.0086 (7)
C9	0.0621 (8)	0.0899 (10)	0.0692 (8)	−0.0047 (8)	0.0297 (7)	0.0048 (8)
C8	0.0740 (10)	0.1048 (12)	0.0621 (8)	0.0107 (9)	0.0288 (7)	−0.0021 (8)
C7	0.0691 (10)	0.1120 (14)	0.0903 (11)	0.0166 (10)	0.0146 (9)	−0.0142 (11)

Geometric parameters (Å, º)

O2—C11	1.2373 (16)	C6—C5	1.379 (2)
C12—C10	1.472 (2)	C6—C1	1.381 (2)
C12—H12A	0.9600	C4—C5	1.378 (2)
C12—H12B	0.9600	C4—H4	0.9300
C12—H12C	0.9600	C5—H5	0.9300
O1—C6	1.3636 (17)	C2—C1	1.380 (2)

O1—C7	1.419 (2)	C2—H2	0.9300
N1—C11	1.3686 (19)	C1—H1	0.9300
N1—C10	1.3790 (17)	C9—C8	1.511 (2)
N1—C9	1.4571 (16)	C9—H9A	0.9700
N2—C10	1.2951 (18)	C9—H9B	0.9700
N2—N3	1.3833 (17)	C8—H8A	0.9700
N3—C11	1.3494 (17)	C8—H8B	0.9700
N3—H3	0.969 (17)	C7—H7A	0.9600
C3—C2	1.379 (2)	C7—H7B	0.9600
C3—C4	1.381 (2)	C7—H7C	0.9600
C3—C8	1.503 (2)		
C10—C12—H12A	109.5	C6—C5—H5	120.0
C10—C12—H12B	109.5	C3—C2—C1	122.24 (13)
H12A—C12—H12B	109.5	C3—C2—H2	118.9
C10—C12—H12C	109.5	C1—C2—H2	118.9
H12A—C12—H12C	109.5	O2—C11—N3	128.53 (14)
H12B—C12—H12C	109.5	O2—C11—N1	127.27 (12)
C6—O1—C7	117.61 (13)	N3—C11—N1	104.19 (12)
C11—N1—C10	107.78 (11)	C2—C1—C6	119.57 (14)
C11—N1—C9	122.61 (11)	C2—C1—H1	120.2
C10—N1—C9	129.44 (13)	C6—C1—H1	120.2
C10—N2—N3	104.65 (11)	N1—C9—C8	113.18 (12)
C11—N3—N2	112.16 (13)	N1—C9—H9A	108.9
C11—N3—H3	123.1 (10)	C8—C9—H9A	108.9
N2—N3—H3	124.7 (10)	N1—C9—H9B	108.9
N2—C10—N1	111.21 (13)	C8—C9—H9B	108.9
N2—C10—C12	124.18 (13)	H9A—C9—H9B	107.8
N1—C10—C12	124.59 (13)	C3—C8—C9	113.98 (12)
C2—C3—C4	117.02 (14)	C3—C8—H8A	108.8
C2—C3—C8	121.17 (14)	C9—C8—H8A	108.8
C4—C3—C8	121.80 (14)	C3—C8—H8B	108.8
O1—C6—C5	115.91 (13)	C9—C8—H8B	108.8
O1—C6—C1	124.82 (14)	H8A—C8—H8B	107.7
C5—C6—C1	119.26 (14)	O1—C7—H7A	109.5
C5—C4—C3	121.91 (14)	O1—C7—H7B	109.5
C5—C4—H4	119.0	H7A—C7—H7B	109.5
C3—C4—H4	119.0	O1—C7—H7C	109.5
C4—C5—C6	119.99 (14)	H7A—C7—H7C	109.5
C4—C5—H5	120.0	H7B—C7—H7C	109.5
C10—N2—N3—C11	0.27 (15)	C8—C3—C2—C1	179.58 (13)
N3—N2—C10—N1	0.27 (15)	N2—N3—C11—O2	177.88 (14)
N3—N2—C10—C12	−178.45 (13)	N2—N3—C11—N1	−0.69 (15)
C11—N1—C10—N2	−0.71 (15)	C10—N1—C11—O2	−177.77 (14)
C9—N1—C10—N2	174.62 (12)	C9—N1—C11—O2	6.5 (2)
C11—N1—C10—C12	178.01 (13)	C10—N1—C11—N3	0.82 (14)
C9—N1—C10—C12	−6.7 (2)	C9—N1—C11—N3	−174.90 (12)

C7—O1—C6—C5	177.44 (12)	C3—C2—C1—C6	−0.4 (2)
C7—O1—C6—C1	−2.57 (19)	O1—C6—C1—C2	179.58 (12)
C2—C3—C4—C5	−0.1 (2)	C5—C6—C1—C2	−0.4 (2)
C8—C3—C4—C5	−179.05 (13)	C11—N1—C9—C8	−85.03 (16)
C3—C4—C5—C6	−0.7 (2)	C10—N1—C9—C8	100.24 (17)
O1—C6—C5—C4	−179.08 (13)	C2—C3—C8—C9	−84.57 (17)
C1—C6—C5—C4	0.9 (2)	C4—C3—C8—C9	94.32 (18)
C4—C3—C2—C1	0.6 (2)	N1—C9—C8—C3	−65.28 (18)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O2[i]	0.969 (17)	1.847 (18)	2.8068 (18)	170.2 (14)
C8—H8B···O2[ii]	0.97	2.57	3.3260 (18)	135

Symmetry codes: (i) −x, −y+3, −z; (ii) −x, y−1/2, −z+1/2.